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मानक

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“Step Out From the Old to the New”

IS 1917-2 (1991): Chemical Analysis of Quartzite and High Silica Sand, Part 2: Determination of Sodium and Potassium By Flame Photometry [MTD 13: Ores and Raw Materials]



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“ज्ञान एक ऐसा खजाना है जो कभी चुराया नहीं जा सकता है”

Bhartrhari—Nitiśatakam

“Knowledge is such a treasure which cannot be stolen”

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भारतीय मानक

उच्चसिलिका बालू एवं क्वार्ट्जाइट का रासायनिक विश्लेषण

भाग 2 ज्वलन प्रकाशमिति पद्धति द्वारा सोडियम तथा पोटेशियम ज्ञात करना

(पहला पुनरीक्षण)

Indian Standard

CHEMICAL ANALYSIS OF QUARTZITE AND HIGH SILICA SAND

PART 2 DETERMINATION OF SODIUM AND POTASSIUM
BY FLAME PHOTOMETRY

(*First Revision*)

UDC 553.57 : 543.24 [546.32+546.33]

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FOREWORD

This Indian Standard (Part 2)(First Revision) was adopted by the Bureau of Indian Standards, after the draft finalized by the Methods of Chemical Analysis of Ores, Minerals and Allied Materials for Metallurgical Industry Sectional Committee had been approved by the Metallurgical Engineering Division Council.

IS 1917 : 1962 Methods of Chemical Analysis of Quartzite and High Silica Sand covers the methods for the determination of silica and other constituents of quartzite and high silica sand. The committee decided to revise this standard into different parts and covering determination of each element in a separate part, which on publication would supersede the determination of that element given in IS 1917 : 1962. The determination of loss on ignition, silica, sodium, potassium, aluminium, iron, calcium, magnesium and titanium oxide in quartzite and high silica sand have been covered in this series.

This part covers the determination of sodium and potassium by flame photometric method. The other parts in this series are:

- Part 1 Determination of loss on ignition
- Part 3 Determination of silica
- Part 4 Determination of aluminium
- Part 5 Determination of iron
- Part 6 Determination of calcium and magnesium
- Part 7 Determination of titania

In reporting the result of a test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS 2 : 1960 'Rules for rounding off numerical values (revised)'.

*Indian Standard***CHEMICAL ANALYSIS OF QUARTZITE
AND HIGH SILICA SAND****PART 2 DETERMINATION OF SODIUM AND POTASSIUM
BY FLAME PHOTOMETRY***(First Revision)***1 SCOPE**

This standard (Part 2) covers the method for determination of alkalis (as NO_2O and K_2O) in the range up to 2.0 percent in quartzite and high silica sand.

2 REFERENCES

The following Indian Standards are necessary adjuncts to this standard.

<i>IS No.</i>	<i>Title</i>
1070 : 1977	Water for general laboratory use (<i>second revision</i>)
1811 : 1984	Methods of sampling foundry sands
2245 : 1962	Methods of sampling quartzite.

3 SAMPLING

3.1 The sample shall be drawn and prepared after quartering in accordance with IS 2245 : 1962 or IS 1811 : 1984, as the case may be.

3.2 Grind 3 to 5 g of the sample so that it passes through IS Test Sieve No. 15 (149 microns). Dry the sample in a weighing bottle at $105^\circ\text{--}110^\circ\text{C}$ for 2 h. Keep the weighing bottle stoppered in a desiccator and cool to room temperature. The sample is ready for chemical analysis.

4 QUALITY OF REAGENTS

Unless specified otherwise, analytical grade reagents and distilled water (*see* IS 1070 : 1977) shall be employed for the test.

5 DETERMINATION OF SODIUM AND POTASSIUM BY FLAME PHOTOMETRY**5.1 Outline of the Method**

After fuming the sample to dryness with hydrofluoric acid and perchloric acid, the residue is dissolved in dilute hydrochloric acid and the solution is diluted to a definite volume. A portion of the solution is atomized in a flame photometer. Concentration of alkalis present are computed by comparing the intensities of their emitted radiations with those of standard solution.

5.2 Apparatus, Flame photometer.

5.3 Reagents

5.3.1 Perchloric Acid, 60 percent (v/v).

5.3.2 Hydrofluoric Acid, 40 percent (v/v).

5.3.3 Dilute Hydrochloric Acid, 1 : 1 (v/v).

5.3.4 Sodium Chloride, Solid.

5.3.5 Potassium Chloride, Solid.

5.3.6 Standard Sodium Solution

Dissolve 0.2541 g of dry sodium chloride in double distilled water and dilute to 500 ml in a volumetric flask (200 ppm Na). Prepare 1,2,3,4 and 5 ppm of standard sodium solution by proper dilution.

5.3.7 Standard Potassium Solution

Dissolve 0.1907 g of dry potassium chloride in double distilled water and dilute to 500 ml in a volumetric flask (200 ppm K). Prepare 1,2,3,4 and 5 ppm of standard potassium solution by proper dilution.

5.4 Procedure

5.4.1 Take 0.1 g of the test sample in a platinum dish, moisten with a few drops of double distilled water and add to it 1 ml of perchloric acid and 5 ml of hydrofluoric acid. Place the dish on sand bath and evaporate till strong fumes of perchloric acid come out. Cool the dish, wash it along with the inner side with double distilled water and add 5 ml of hydrofluoric acid. Repeat the process twice and evaporate on sand bath to just dryness.

5.4.2 To the mass 5.4.1 add 5 drops of dilute hydrochloric acid (1 : 1) and 25 ml of water. Place the dish on steam bath. After 5 to 10 minutes, transfer quantitatively the contents to a 100 ml volumetric flask and dilute up to the mark with double distilled water.

5.5 Determination of Sodium

5.5.1 Place a few ml of the test solution (5.4.2) in the cup of a flame photometer. Measure the emission intensity at 589 nm or through a sodium

filter against double distilled water.

5.5.2 Draw the calibration graph with 0,1,2,3,4 and 5 ppm concentration of standard sodium solutions. Calculate the unknown sodium concentration by referring to this graph.

5.5.3 Calculate the percentage of sodium content as follows:

$$\text{Sodium, percent by mass} = \frac{A \times 100}{B \times 10^6}$$

where

A = concentration in ppm, of sodium; and

B = mass in g, of the sample represented by the aliquot taken in the final test solution.

5.5.4 From the value of the sodium content as obtained in 5.5.3, calculate the percentage of sodium oxide as follows:

$$\% \text{ Na}_2\text{O} = \% \text{ Na} \times 1.348$$

5.6 Determination of Potassium

5.6.1 Place a few ml of the test solution (5.4.2) in the cup of the flame photometer. Measure the emission intensity at 767 nm or through a potassium filter against double distilled water.

5.6.2 Draw a calibration graph with 0,1,2,3,4 and 5 ppm of standard potassium solutions. Calculate the unknown potassium concentration by referring to this calibration graph.

5.6.3 Calculate the percentage of potassium content as follows:

$$\text{Potassium, percent by mass} = \frac{C \times 100}{D \times 10^6}$$

where

C = concentration in ppm, of potassium, and

D = mass in g, of the sample represented by the aliquot taken in the final test solution.

5.6.4 From the value of potassium content as obtained from 5.6.3, calculate the concentration of potassium oxide as follows:

$$\% \text{ K}_2\text{O} = \% \text{ K} \times 1.205$$

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